EMLab P&K

Title: Asbestos Analysis in Soils and Rock: CARB 435 using PLM

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Approvals

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Regional Laboratory Director, West	**Regional Quality Assurance Manager, Central**

1.0 SCOPE AND APPLICATION

1.1 The following procedure describes the protocol for CARB 435 and point counting technique using PLM for analyzing soils and rock.

1.2 The standard CARB 435 method includes 400 point counting for quantification of asbestos fibers when detected. However, based on client's request either 200 or 1000 point counting may also be performed.

1.3 The analyses will be performed in accordance with the California Air Resources Board (CARB) Method 435 for the determination of asbestos in serpentine aggregate samples, adopted on June 6, 1991.

1.4 This method is applicable to determining asbestos content of serpentine aggregate in storage piles, on conveyor belts, and on surfaces such as roads, shoulders and parking lots.

1.5 The analysis must only be performed at laboratories equipped with a HEPA filter for handling and processing the submitted samples.

2.0 SUMMARY

2.1 Sample preparation follows a standard CARB 435 prep method. If crushing of the sample is required, then the sample will be sent to an offsite location to be crushed. The entire sample will be dried at 135-150°C and then crushed to ~3/8" gravel size. The sample will be pulverized to produce a nominal 200 mesh final product.

2.2 Small aliquots of the sample will be mounted on three separate microscope slides containing the appropriate refractive index oil and analyzed by PLM with EPA Method 600/R-93/116. If asbestos is identified and is detected to be less than 10% concentration by visual area estimate then additional sample mounts are prepared as needed for performing point counting (400 point counts or 1000 point counts) and the quantification of asbestos concentration will be obtained using the standard CARB Method 435 point count protocol.

3.0 DEFINITIONS

3.1 **Asbestos**. A family of naturally occurring minerals, found in serpentine and other metamorphic rock. Refers to the asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-grunerite); anthophyllite; tremolite; and actinolite.

3.2 **ACM.** Asbestos Containing Materials are any materials or products that contain more

than one percent asbestos.

3.3 **Detection Limit.** The number of fibers necessary to be 95% certain that the result is greater than zero.

3.4 **Fiber**. A particle that is 5 micrometer or longer, with a length-to-width ratio of 3 to 1 or longer.

3.5 **Friable materials**. Materials that can be crumbled, pulverized, or reduced to powder by hand pressure. This includes non-friable material that becomes damaged to the extent that when it is dry, it may be crumbled, pulverized, or reduced to powder by hand pressure.

3.6 **H.D.** "High Dispersion" Refractive Index

3.7 **SACM**. Suspected Asbestos Containing Material. All bulk samples received in the laboratory for any type of analysis will be considered SACM. The term SACM has long been used by the asbestos industry to refer to any building material that is suspected of being asbestos-containing but has not been proven conclusively to be ACM (based on sampling and analysis, documentation, building records, etc). Suspect material would include any material suspected of containing asbestos and is found in a building constructed after 1980, or any material found in a building constructed prior to 1981."

3.8 **PLM**. Polarized Light Microscopy.

3.9 **R.I.** Refractive Index -The ratio of the velocity of light in a vacuum to the velocity of light in a medium.

3.10 **TAT.** Turn Around Time: The span of time in which the laboratory completes the analysis of a project, or portion of the project, from sample receipt to reporting of data.

3.11 **Serpentinite**: Serpentine containing rock or soil.

3.12 **Grab sample**. A sample taken from a volume of material.

3.13 **Composite sample**. A mixture or blend of material taken from more than one grab sample.

4.0 INTERFERENCES

4.1 There are no known interferences to this method.

5.0 SAFETY

5.1 General

5.1.1 Inspect and wear appropriate protective clothing/equipment as procedure dictates and when necessary to avoid exposure.

5.1.2 All sample handling and examination must be carried out in a HEPA-filtered hood with continuous negative air flow. Handling samples without these precautions may result in exposure to airborne fibers.

5.1.3 Assume all bulk samples received in the laboratory for analysis to have asbestos and handle them accordingly. Never open or remove a PACM from the zip lock bag/container unless it is in a HEPA filtered hood.

5.1.4 Wet wipe the interior of all PLM hoods and stereoscopes before turning them off at the end of each use. Also vacuum the hoods with the HEPA vacuum on a frequent basis (daily or weekly).

5.1.5 Wet wipe down all work surfaces, such as the fume hood and laboratory bench areas, before and after use.

5.1.6 Wash all areas of exposed skin prior to leaving the laboratory.

5.2 Primary Materials Used - Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS

Material	Hazards	Exposure Limit	Signs and Symptoms of exposure
Common Commercial Asbestos (Chrysotile	Carcinogenic; eye, skin, lung, and	0.1 fibers/cc	Asbestos corns on skin. Respiratory tract irritation. Cough and chest pain. Chronic exposure: asbestosis, interstitial fibrosis of the lung tissue, respiratory or cardiac failure, secondary lung infections, pleural effusion,

, Amosite, Crocidolite)	gastroint estinal irritant		mesothelioma, lung cancer, bronchogenic carcinoma, cancers of the gastrointestinal tract and larynx.
Cargille R.I. liquid 1.550	None known	N/A	Oily feel. Eye irritation
Cargille R.I. liquid 1.605	None known	5 mg/cubic meter (OSHA PEL)	Oily feel. Red welt on skin of sensitive individuals. Eye irritation
Cargille R.I. liquid 1.680	None known	5 mg/cubic meter (OSHA PEL)	Oily feel. Headaches and nausea possible. Eye irritation

6.0 EQUIPMENT / INSTRUMENTS

6.1 HEPA ventilated, negative pressure hood with an air flow rate of at least 75 fpm at the opening

6.2 Hot plate

6.3 Lab tools/supplies:
- **6.3.1** 70% Isopropyl Alcohol
- **6.3.2** Cover slips, 22mmx22mm or 18mmx18mm, thickness 1.5
- **6.3.3** Dissecting needles
- **6.3.4** Forceps
- **6.3.5** Glass plate
- **6.3.6** Kim wipes, paper towels
- **6.3.7** Microscope slides
- **6.3.8** Mortar and pestle: agate or porcelain
- **6.3.9** Scalpel
- **6.3.10** Spatula
- **6.3.11** Tweezers

6.4 Polarized Light Microscope which includes:
- **6.4.1** Sub-stage polarizer that can rotate
- **6.4.2** Analyzer oriented perpendicular to polarizer
- **6.4.3** Port for retardation plate
- **6.4.4** 360° graduated rotating stage with 1° increments
- **6.4.5** Sub-stage condenser with centerable iris
- **6.4.6** Lamp, and lamp iris
- **6.4.7** Objective lenses: 10X, 20X, and 40X or near equivalent

6.4.8 Ocular lens: 10X minimum.
6.4.9 Eyepiece reticule: cross hair.
6.4.10 Dispersion staining objective lens
6.4.11 Compensator plate/Wave plate: 550 nm± 20 nm, retardation "first order red" compensator
6.4.12 Orange filter (to determine Becke line)
6.4.13 Chalkey point graticule or cross point Graticule
6.4.14 Bertrand lens or a telescope ocular to view the central stop

6.5 Reference materials (where available or equivalent):
6.5.1 NIST SRM 1866a (common) for Chrysotile, Grunerite (Amosite), Riebeckite (Crocidolite) and Glass Fiber
6.5.2 NIST SRM 1867 (uncommon) for Anthophyllite, Tremolite, and Actinolite
6.5.3 Reference book on mineralogy and/or crystallography

6.6 Stereomicroscope, Low power, minimum of 10X magnification

6.7 Room thermometer that is verified annually against a NIST-traceable standard, (per SOP EM-EQ-S-1584, Equipment Calibration and Maintenance)

6.8 Vacuum, HEPA-filtered

7.0 MEDIA AND REAGENTS

7.1 Refractive index (RI) liquids for Dispersion Staining: High-Dispersion (HD) series: 1.550, 1.605, 1.625, 1.630, 1.640, 1.680, and 1.700.

7.2 Series of refractive index oils that have refractive indexes between 1.49 and 1.72 in intervals less than or equal to 0.005.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests.

8.1.1 This method does not have any referenced holding time.

9.0 QUALITY CONTROL

9.1 Contamination checks will be performed each day of operation and recorded in the PLM Daily and Monthly QA Checklist.

9.2 Refractive index (RI) liquids used will be calibrated using glass standards per

Document EM-AS-S-1263, "Refractive Index Calibrations". Reference document EMAS-S-1263 for frequency and documentation requirements. These checks shall be recorded in the PLM Daily/Monthly QC checklist or similar document.

9.3 The manufacturer's instructions for illumination, condenser alignment and other microscope adjustments for the Polarized Light Microscope will be followed for daily calibrations. These adjustments must be performed at the beginning of each day of analysis and results shall be recorded in the PLM Daily and Monthly QA Checklist.

9.4 The air flow rates of the PLM HEPA filtered hoods will be measured on a regular basis. The air flow rate should not be less that 75 fpm at the opening. Calibrations shall be recorded in the PLM Daily and Monthly QA Checklist.

9.5 The room thermometer will be verified against a NIST-traceable standard annually within a temperature range of 20°C to 30°C (per SOP EM-EQ-S-1584, Equipment Calibration and Maintenance).

10.0 PROCEDURE

10.1 First, check the chain of custody to confirm the test to be performed. Pay attention to any special procedures, instructions, or deviations requested by the customer.

10.2 Check that all samples listed on the Chain of Custody (COC) form are present and that all the client sample identification information on the bag matches the information on the COC. Check to make sure that all samples are friable samples. Perform this check underneath the HEPA filtered hood with the blower on. If a discrepancy is noted, then let the project manager associated with the project know to inform the client.

10.3 If any of the zip lock bags are ripped, opened, or not sealed properly, enter a note in the project log indicating that the bag was opened and that there may be possible cross-contamination of other samples. Also, inform the project manager.

10.4 Put the sample bags in the order in which they appear on the COC.

10.5 Sample Preparation: Sample must be prepped within a fume hood, equipped with a HEPA filter.

10.5.1 Quantification of Asbestos Content

10.5.1.1 Visual Method: The standard operation procedure of the visual technique allowed in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program,

Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 dated October 1988.

a. Prepare three slides.

b. View 10 fields per preparation. Identify all fibers.

Exception I: If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. If all fibers are non-asbestos, report "Point counting as stated under "Total Points Counted" was not performed as no asbestos fibers were detected."

Exception II: If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos.

c. If one fiber is determined to be asbestos, discontinue the visual method and perform the point counting technique as described below.

10.5.1.2 Point Counting Rules

a. Prepare preparations with the appropriate refractive index liquid of the representative sample. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. A maximum of 100 points will be counted per prep. Thus, a minimum of 10 preps will be made for a 1000 point count, 4 for a 400 point count and 2 for a 200 point count.

b. Record the number of points positioned directly above each particle or fiber.

c. Record only one point if two points are positioned over same particle or fiber.

d. Record the number of points positioned on the edge of a particle or fiber.

e. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on the visual intersection, a point is scored for both categories.

f. If a test point lies over an ambiguous structure, no particle or fiber is recorded.

g. A fiber mat or bundle is counted as one fiber.

h. Asbestos fibers are defined as mineral fibers having an aspect ratio greater than 3:1.

11.0 CALCULATIONS

11.1 Calculate the asbestos percentage with the following equation

% asbestos = a / t * 100

a = total number of asbestos points counted
t = total number of occupied points counted

For example if 400 occupied points were observed and 20 asbestos fibers were counted then:

% asbestos = 20 / 400 * 100
% asbestos = 5%

11.2 Reporting Results

11.3 The asbestos and non-asbestos fiber concentration in the sample will be reported as a percentage.

11.4 If the customer requires a deviation from the documented standard operating procedure, these must be entered into the report comment section and project log, and must be communicated to the appropriate personnel.

12.0 METHOD PERFORMANCE

12.1 Methods stated in the SOP are validated continuously through inter and intra analyst comparisons, duplicate analysis, proficiency samples, daily reference slides, and routine quality control checks.

12.2 Employees must abide by the training policies and procedures in the Quality Assurance Manual, SOP EM-AS-S-1261 "Asbestos Analysts Training" and this document.

13.0 HAZARDOUS WASTE DISPOSAL

13.1 Wet wipe all areas under the PLM HEPA-filtered hoods with water or alcohol. Dispose of paper towel/kim wipe into asbestos waste. After wiping, vacuum all hoods with the HEPA vacuum before turning the hood off at the end of the day or at the end of each use.

13.2 Wipe down work surfaces, such as the laboratory bench areas, before and after use with a paper towel/Kim wipe and water or alcohol. Dispose of paper towel/kim wipe into asbestos waste.

13.3 Double bag and store all asbestos waste in a designated area for a period of 30 days or greater (per SOP EM-HS-S-1286, Sample Retention and Disposal) and appropriately dispose of if no special request for holding has been made by the client. Separately place projects that have at least one sample with asbestos detected at any quantity in a separate bin and classify them as ACM for regulated disposal. Classify all projects with no samples containing asbestos as NACM and dispose them in regular trash.

13.4 Dispose of expired reagents according to the manufacturer's instructions. See Safety Data Sheet for proper disposal.

13.4.1 Safety Data Sheets (SDS) can be found in designated areas in each of EMLab P&K Laboratory facilities or accessed online.

14.0 REFERENCES/CROSS-REFERENCES

14.1 Perkins, R.L., and Harvey, B.W., Test Method: Method for the Determination of Asbestos in Bulk Building Materials. EPA 600/R-93/116. Washington. D.C. July 1993

14.2 National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 dated October 1988.

15.0 ATTACHMENTS

15.1 None

14.0 REVISION HISTORY

15.2 Revision 00, dated 10/30/2009

15.2.1 Integration for TestAmerica Facilities

15.3 Revision 01, dated 6/13/2012

15.3.1 Revised into new Analytical SOP template
15.3.2 Included definitions for PACM and RI.
15.3.3 Removed Sampling Instructions
15.3.4 Referenced Doc 1263, Refractive Index Calibrations and Doc 1584, Calibration and Maintenance of Lab Equipment in the QA section.

15.4 Revision 02, dated 03/20/15
15.4.1 Updated into new SOP template and reference document numbers
15.4.2 Added the statement "where available or equivalent" after the reference materials.
15.4.3 Updated MSDS (Material Safety Data Sheet) to SDS (Safety Data Sheet)

15.5 Revision 03, dated 03/30/17
15.5.1 Updated document owner on cover page.
15.5.2 Updated MSDS (Material Safety Data Sheet) to SDS (Safety Data Sheet) in section 5.2
15.5.3 Revised RI Indices calibration frequency and procedure in section 9.2
15.5.4 Revised procedure to monitor air flow rates in section 9.4
15.5.5 Updated procedure for exception I method in section 10.5.1.1